METHOD AND SYSTEM FOR CONTINUOUS IN SITU MONITORING OF VISCOSITY

This is a continuation of copending application Ser. No. 07/161,351 filed on Feb. 22, 1988; now abandoned which is a continuation of copending application Ser. No. 806,493 filed on Dec. 9, 1985, now abandoned.

BACKGROUND AND SUMMARY OF THE INVENTION

This invention relates to a method and a system for monitoring viscosity and in particular to a fiber-optic system for continuous in situ monitoring of viscosity by monitoring viscosity-dependent fluorescence.

There are well known laboratory techniques for measuring viscosity, but these have limited application. There are many instances where it would be desirable to continuously monitor the viscosity of a substance in situ. For example it would be desirable to continuously monitor the viscosity in chemical processes to monitor the extent or the rate of chemical reactions. In a polymerization or resin cure process monitoring of viscosity would indicate the extent of reaction i.e. the degree of cure and thus permit greater control of the process and the resultant product. It would also be desirable to continuously monitor the viscosity of other substances such as lubricating oils, insulating oils, and hydraulic fluids to detect breakdown.

The present invention is a new method and system for the continuous in situ monitoring of the viscosity of a substance. The system monitors the viscosity of the substance by monitoring its fluorescence. Fluorescence is the emission of energy in the form of visible light in response to excitation or activation of the substance by radiation. Certain substances exhibit viscosity-dependent fluorescence, that is, the fluorescence of these substances is dependent upon the viscosity of the medium in which they are dissolved. Some of the substances that will be monitored exhibit viscosity-dependent fluorescence themselves; other substances can be doped with a viscosity-dependent fluorescent substance. Thus fluorescence can be used to measure viscosity and degree of cure.

It is generally accepted that in viscosity-dependent fluorescent substances the energy absorbed from external irradiation is emitted through fluorescence and through intramolecular torsional and twisting motions. In a low viscosity medium a substantial amount of the energy can be emitted through these intramolecular torsional and twisting motions resulting in low fluorescence yield. However, as the viscosity of the medium increases intramolecular torsional and twisting motions become progressively more inhibited, resulting in a gradual increase in fluorescence.

The level of fluorescence and thus the viscosity is measured by fiber-optic fluorometry. Fiber optic waveguides are used to transmit excitation energy, typically ultra violet light to the substance from a remote source. The same or different waveguides also transmit the resultant fluorescence from the substance to remote processing instruments. The fiber optic waveguides allow in situ monitoring at a location remote from the light source and processing instruments. Processing of the resultant fluorescent light includes isolating the wave lengths of interest with beam splitters and filters or with monochromators and measuring their intensities with photodiodes or photodiode arrays. The analog signals from the photodiodes can then be amplified, digitalized, and interpreted. Such interpretation may include comparison with a predetermined calibration table of corresponding viscosities, which can conveniently be done with a microprocessor.

The inventor's new method and system permits the continuous, in situ monitoring of the viscosity of a substance. The method and system achieve accurate monitoring at a relatively modest cost. The use of fiber optic waveguides allows in situ monitoring at locations remote from the monitoring equipment, such as inside reaction vessels or large autoclaves. The viscosity of the substance can thus be continuously monitored during a reaction or while in service. The fiber optic waveguide does not even have to be in physical contact, just optical contact, with the substance.

The inventor's method and system is particularly useful for the monitoring and control of the manufacture of composite materials. The properties of composite materials, for example carbon-epoxy composites, depend upon the chemical and rheological events occurring during the resin cure cycle. Thus the properties of a composite material can be controlled by controlling the cure cycle of the composite material. The ability to control the cure cycle depends upon the ability to monitor the polymerization process of the composite material as the resin cures. Thus an improvement in monitoring improves the control of the cure cycle and allows the process to approach the optimum conditions to maximize the desired properties of the composite and allows a greater degree of control over the end product. The ability to conduct continuous in situ monitoring of resin cure states, cure kinetics, and resin viscosity greatly enhances control over the mechanical properties of composite materials.

Rheological, thermoanalytical, and spectroscopic techniques that measure cure variables have been used in laboratory studies of resin cure behavior. Physical restrictions, however, prevent the application of these techniques to in situ measurements during curing of composite materials in a factory setting. Dielectric spectroscopy techniques have been the only commercially acceptable method of monitoring cure viscosity. Senturia, et al. have recently described a microdielectrometric method for measuring cure viscosities. See "In-situ Measurement of the Properties of Curing Systems with Microdielectrometry," *Journal of Adhesion* 15, p. 69 (1982), incorporated herein by reference.

Fortuitously, some resins used in composite materials exhibit viscosity-dependent fluorescence. For example, tetraglycidyldiaminodiphenylmethane epoxy, which is the main constitutent of the widely used CIBA-GEIGY MY720 (TM) resin, exhibits viscosity-dependent fluorescence. Other resins, for example those based on diglycidyl ether of bisphenol A do not exhibit viscosity-dependent fluorescence. However, small amounts (in the range of about 0.1–0.5%) of a viscosity-dependent fluorescent material can be added to these resins.

The inventor's method and system permits continuous in situ monitoring of resin viscosity or the degree of cure by monitoring the fluorescence of the resin. This continuous monitoring applied to the cure of the resin in a composite material allows greater control over the cure process and thus greater control over the properties of the resulting composite. The continuous monitoring allows the cure process to be conducted to maximize the properties of the composite. The system achieves accurate monitoring at a relatively modest